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UTAH UNIV SALT LAKE CITY DEPT OF CHEMISTRY
STUDIES OF ELASTIC CONSTANTS OF ORIENTED POLY (ETHYLENE TEREPHT--ETC(U)
MAY 81 D B CAVANAUGH, C H WANG

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REPORT DOCUMENTATION PAGE		READ INSTRUCTIONS BEFORE COMPLETING FORM
1. REPORT NUMBER	2. GOVT ACCESSION NO.	3. RECIPIENT'S CATALOG NUMBER
Technical Report #4	AD-A100 313	
4. TITLE (and Subtitle)	5. TYPE OF REPORT & PERIOD COVERED	
Studies of Elastic Constants of Oriented Poly (Ethylene Terephthalate) Films Using Brillouin Scattering.	<input checked="" type="checkbox"/> Technical Report	
7. AUTHOR(s)	6. PERFORMING ORG. REPORT NUMBER	
David B. Cavanaugh and C. H. Wang	N00014-79C-0507 Serial RC-607	
9. PERFORMING ORGANIZATION NAME AND ADDRESS	10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS	
Department of Chemistry University of Utah Salt Lake City, Utah 84112		
11. CONTROLLING OFFICE NAME AND ADDRESS	12. REPORT DATE	
Office of Naval Research 800 N. Quincy Street Arlington, Va. 22217	May 21, 1981	
14. MONITORING AGENCY NAME & ADDRESS (if different from Controlling Office)	13. NUMBER OF PAGES	
	22	
16. DISTRIBUTION STATEMENT (of this Report)	15. SECURITY CLASS. (of this report)	
Approved for public release; distribution unlimited	Unclassified	
17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)	15a. DECLASSIFICATION/DOWNGRADING SCHEDULE	
18. SUPPLEMENTARY NOTES		
Prepared for publication in Applied Physics		
19. KEY WORDS (Continue on reverse side if necessary and identify by block number)		
Poly(ethylene Terephthalate) Brillouin Scattering Stretched Film Longitudinal and Transverse Acoustic Phonone Velocities Elastic Constants	Orientation	
20. ABSTRACT (Continue on reverse side if necessary and identify by block number)		
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Serial RC-607

Technical Report No. 4

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FILMS USING BRILLOUIN SCATTERING

By

David B. Cavanaugh & C. H. Wang

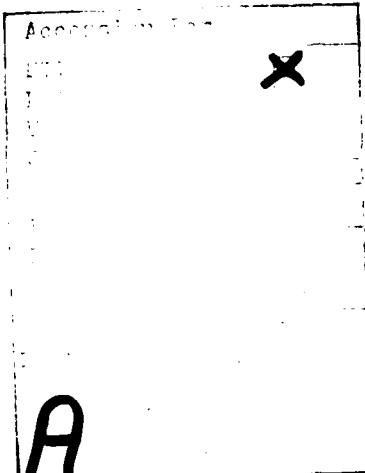
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Prepared for Publication
in
Applied Physics

May 21, 1981

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FILMS USING BRILLOUIN SCATTERING

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ABSTRACT

Brillouin scattering is used to study the elastic behavior of oriented poly(ethylene terephthalate) films. The elastic constants C_{11} , C_{13} , C_{33} and C_{44} are determined by measurements of the quasi-longitudinal and quasi-transverse hypersonic velocities at different angles in the film. The total orientational parameter is calculated for the three draw ratios from the Brillouin hypersonic velocities and are found to be similar to the orientational parameter in the amorphous phase obtained with a different technique.

INTRODUCTION

It is well known that the elastic properties of polymers are affected by externally applied perturbations such as stretching or extrusion. The external perturbation induces the polymer chains to assume a preferred alignment in the sample relative to the axis of deformation. This alignment increases the chain orientation in both the crystalline and amorphous regions. One manifestation of this orientation is an increase in the elastic stiffness along the axis of alignment.

Polyethylene terephthalate (PET) is a crystalline polymer when it has been cooled slowly from the melt. It is an amorphous polymer when quenched suddenly from the molten state. Amorphous films of PET are known to develop stress induced crystallinity during the process of orientation. (1) This crystallinity causes a radical alteration of the superstructure in the oriented polymer from that in the amorphous film. It is therefore useful to see how the elastic constants of PET films are affected by orientation.

Brillouin Light Scattering is a probe of the hypersonic acoustic phonons in a given medium. Information about the hypersonic velocities and acoustic attenuation is readily obtained by Brillouin scattering while the medium remains close to thermodynamic equilibrium. With the development of the multipass Fabry Perot interferometer it is now possible to observe Brillouin scattering in media even with strong elastic scattering from surfaces or inclusions. Further, a five pass interferometer provides sufficient contrast that the transverse phonons in polymer films of

moderate thickness can be detected.

The hypersonic velocity, v_B , in an optically isotropic medium is related to the Brillouin frequency shift, f_B , by:

$$v_B = \frac{f_B \lambda_i}{2 n \sin \theta/2} \quad (1)$$

where λ_i is the incident wavelength, n is the refractive index, and θ is the scattering angle. In a birefringent medium Eq. (1) becomes more complicated.

Advantages for using Brillouin scattering for polymer work are that the size and shape of the sample are relatively unimportant, and this reduces the amount of preparation necessary for a measurement. In addition, the phonon propagation direction in the sample is accurately defined by the scattering geometry. In this paper we report the effect of orientation on the elastic constants of PET films subjected to uniaxial orientation as determined with Brillouin scattering.

EXPERIMENTAL

PET was purchased from the Aldrich Chemical Company as amorphous nuggets. Films were cast in a hydraulic press fitted with polished heated plates. The cast temperature was 270°C and 2 metric tons of pressure was applied for 10 minutes. The hot films were quenched in ice water. The strongly adhering aluminum foil jacket was removed by (2) dissolving in 1 M NaOH. Regions of the films having an unblemished surface and uniform thickness were selected for experimentation.

The films were uniaxially oriented in a water bath at 80°C using a manually operated film puller. The draw rate was about 100%/min. The final elongation was determined by measuring the separation of marks on the film before and after stretching. A stretch ratio of 4.66 was found to be just less than the break point at this temperature. The films were mounted in frames directly from the puller so that their final elongation was maintained. Annealing the PET films in heated baths tended to reduce the optical quality by making the samples less transparent; therefore, the films used in this experiment were unannealed.

A triple pass Fabry Perot (F.P.) was used in the first stage of the (3) experiment. A five pass F.P. is used in the present work to obtain the various elastic constants. The finesse of the 5 pass F.P. is above 60. The F.P. mirrors are scanned piezoelectrically and the free spectral range used in this experiment is about 30 GHz.

An argon ion laser^o tuned to 4880 Å and equipped with an etalon provides

the incident radiation. The photo-current from the photomultiplier tube is amplified with a picoammeter and the spectra are displayed on a chart recorder. The beam is focused to a small area on the film by a lens of 5 cm focal length. The incident radiation is polarized vertically by the laser and a polarization analyzer selects the vertical component of the scattered light so that all spectra are polarized.

The film thickness varies slightly from 0.014 cm for the unoriented to 0.011 cm for the highest stretch ratio. The refractive index of the films was determined by finding the critical angle of total reflectance at the base of a prism with a layer of high index liquid placed between the prism base and the film. The index of the unoriented films is found to be 1.588 at 4880 \AA . The film densities were determined with a density gradient column. The films vary in density with draw ratio. The degree of crystallinity in the unoriented film is less than 8%.

The angular measurements for the velocity contours were made with a goniometer as shown in Fig. 1a. The goniometer was adjusted so that the rotation axis about α is in the scattering plane. The film was mounted so that a rotation about α rotates the film sample about its Y axis (Fig. 1b). The scattering region of the film was positioned to remain in the center of rotation as α is varied. α is defined to be the angle between the film stretch axis and the scattering vector. The angle β was fixed so the incident and scattered light beams make an angle of 45° to the film surface.

Two sets of Brillouin doublets are found in the polarized spectrum of the amorphous film. A strong band is seen at 6.9 GHz and a second weaker band is found at 15.6 GHz. No peaks are observed in the depolarized scattering spectrum of these films, despite the fact that depolarized spectral peaks have been reported in PET films.^{4,11} The 90° scattering geometry in

Fig. 2 shows two incident light rays from which Brillouin scattering occurs: the first is from the incident beam as it makes the first pass through the film, and the second is due to the reflected beam from the back surface of the film. Thus for the 90° scattering there are two scattering vectors: q_1 which lies in the plane of the film, and q_2 which is tilted ⁵ from the normal direction of the film.

The spectra obtained in the 90° scattering geometry have an interesting feature. When the film bisects the angle made by the incident and scattered beams, the sound velocity measured from the scattering vector ⁵ (q_1) is independent of the refractive index of the medium. In this case the sound velocity is given simply by:

$$v_s = \frac{f_B \lambda_i}{\sqrt{\frac{2}{2}}} \quad (2)$$

This result is very useful in studying films for which the index of refraction is difficult to measure.

Although there is a large difference in Brillouin frequency for peaks associated with q_1 and q_2 , the sound velocities are found to be the same in the unoriented sample, and suggest that the sample is isotropic and there is no dispersion with wave vector.

RESULTS AND DISCUSSION

Figure 3 shows Brillouin spectra of oriented PET films at $R_s = 3.57$. When the oriented films are rotated about their Y axis to angles above $\alpha > 10^\circ$, a band associated with the quasitransverse (QT) phonon appears at low frequency

in the spectrum. The QT and QL phonon peaks are indicated by (1) and (3), respectively in Fig. 3. The peak indicated as (4) is due to back scattering from the reflected beam, and that indicated as (2) is anomalous and is designated the Y-band. The intensity of the QT band increases up to an angle of about $\alpha \sim 45^\circ$ where it reaches a maximum. The relative intensities of the QL and QT scattering at $\alpha = 45^\circ$ changes with draw ratio. The QL peak is more intense than the QT in the $R_s = 2.68$ film. However, in the $R_s = 4.66$ film, the QT peak is nearly twice the intensity of the QL. The QT scattering fades from the spectrum above $\alpha > 75^\circ$. The QL scattered intensity remains nearly constant up to an angle $\alpha \sim 60^\circ$ where it begins to increase dramatically. It reaches a maximum intensity at $\alpha = 90^\circ$. The positions of various peaks also change with the angle α .

Figure 4 shows a set of polar plots of sound velocity versus the propagation direction in the films. The sound velocity along the z (stretch) axis increases considerably with stretch ratio, which is an expected result as the polymer chains assume a preferred orientation in the stretch direction. The sound velocity along the x axis (perpendicular to stretch) is relatively unaffected by the orientation. The transverse velocities are also affected by stretch. They are found to be smaller in the $R_s = 2.68$ film than in the $R_s = 3.57$ and 4.66 films in which the velocities and contours are similar.

We assume that a uniaxially stretched film has a uniaxial elastic symmetry. The elastic constant matrix for uniaxial symmetry is:

$$c_{ij} = \begin{pmatrix} c_{11} & c_{12} & c_{13} & 0 & 0 & 0 \\ c_{11} & c_{13} & 0 & 0 & 0 & 0 \\ c_{33} & 0 & 0 & 0 & 0 & 0 \\ c_{44} & 0 & 0 & 0 & 0 & 0 \\ c_{44} & 0 & 0 & 0 & 0 & 0 \\ c_{66} & 0 & 0 & 0 & 0 & 0 \end{pmatrix} \quad (3)$$

where $c_{66} = \frac{1}{2} (c_{11} - c_{12})$. From the Cristoffel equation for uniaxial symmetry one can obtain the eigenvalues for the QL (+root) and QT (-root) phonon. They are given by:

$$\begin{aligned} 2 \rho V^2 \pm &= (c_{11} \ell_x^2 + c_{33} \ell_z^2 + c_{44}) \pm \{ (c_{11} \ell_x^2 + c_{33} \ell_z^2 + c_{44})^2 \\ &- 4[(c_{11} \ell_x^2 + c_{44} \ell_z^2)(c_{44} \ell_x^2 + c_{33} \ell_z^2) \\ &- \ell_x^2 \ell_z^2 (c_{13} + c_{44})^2] \}^{\frac{1}{2}} \end{aligned} \quad (4)$$

where: $\ell_x = q_x/|q| = \sin \alpha$ and $\ell_z = q_z/|q| = \cos \alpha$, are the direction cosines of the scattering vector in the film. To obtain the elastic constants c_{11} , c_{33} , c_{44} , and c_{13} , we used a least squares fitting program to find the best fit of the data to Eq. 4. The fitted velocity contours are shown in Fig. 4. The elastic constant c_{66} which would be found from the third eigenvalue of the Cristoffel equation cannot be determined with Brillouin scattering since the transverse acoustic wave associated with c_{66} is not active in Brillouin scattering for the uniaxial symmetry. This is consistent with the experiment.

The elastic constants obtained in this manner are plotted versus draw ratio in Fig. 5. It is clear that the elastic constant most affected by orientation

is C_{33} which increases linearly with stretch ratio. C_{13} also increases with stretch ratio while C_{11} decreases slightly. C_{44} remains unaffected by the film orientation.

The elastic modulus along the orientation axis of the polymer samples are often determined with low frequency sonic velocity measurements. Moseley has proposed a simple theory to determine from the elastic modulus the Hermann orientation parameter, f , according to the expression given by:⁷

$$f = \frac{3}{2} \cos^2 \theta - 1 = 1 - \left(\frac{C_{33}^0}{C_{33}} \right) \left(\frac{\rho}{\rho_0} \right) \quad (5)$$

In this equation θ is the angle between a polymer chain segment and the orientation axis. C_{33}^0 and ρ_0 are the elastic constant and the density of the unoriented sample, respectively.

The Hermann orientation parameter determined according to Eq. (5) is plotted versus draw ratio in Fig. 6. Also plotted in Fig. 5 are the data from Ref.(8) in which the orientation parameter of the amorphous phase isolated from the total orientation parameter is given. Clearly one sees that both sets of data approach a limiting value of $f \approx .75$ at high draw ratio. Since the Brillouin result should correspond to a measure of the total orientation function where the crystalline and amorphous regions transmit acoustic waves cooperatively, the similarity of the Brillouin scattering result to that of the purely amorphous phase orientation function suggests that the factor most affecting the sound velocity in oriented PET is probably due to the orientation of the amorphous

phase chains.

It has been shown previously that the longitudinal Brillouin band splits into two peaks when the film is oriented. We have taken the peak indicated as (3) in Fig. 2 to be scattering from the longitudinal phonon. The second peak, denoted as the Y-band, behaves differently than the longitudinal band when the angle α is varied. Fig. 7 is a polar plot of frequency shift versus α for the QL band and the Y-band in the $R_s = 3.57$ film. At $\alpha = 0$ the Y-band is lower in frequency than the quasilongitudinal band. The two bands merge at $\alpha = 30^\circ$ and when $\alpha > 45^\circ$ the Y-band is found at higher frequencies than the QL band. As α approaches 90° the Y-band intensity is faint and the linewidth appears to be very broad.

Other researchers have found anomalous bands in the Brillouin spectra of oriented polymers. For example, the workers in Ref. (9) have reported a high frequency band which they have attributed to scattering from void defects in oriented Poly(methyl methacrylate). It appears more likely that this is due to scattering from an internal reflection of the incident beam from the back surface of the sample. A simple calculation taking in account the different scattering angles of the incident and reflected beams and the anisotropic sound velocity shows this to be reasonable. However, the anomalous bands in the spectra of oriented PET films and extruded rods¹⁰ cannot be explained in terms of the scattering from a reflected beam. PET is known to develop stress induced crystallinity when it is oriented.⁽¹⁾ The size of the crystalline regions formed in this way can have a size on the order of the hypersonic wavelength and the anomalous band could be the scattering from those regions. However, an anomalous band having similar

features is also seen in the Brillouin spectrum of oriented Poly(propylene) films in which stress induced crystallinity is not present. Further work to elucidate the nature of this work is still in progress.

SUMMARY

Brillouin light scattering is used to obtain the elastic constants of oriented PET films. The elastic constant C_{33} is found to be most affected by the deformation. The sound velocity data are fit to the Christoffel equation of linear elastic theory using a least squares fitting routine. The elastic constants were obtained by fitting the experimental sound velocities obtained at different angles in the films to the theory. The Hermann orientation parameter for each stretch ratio is obtained from the hypersonic velocity data. These are found to be similar to the Hermann parameter calculated for the amorphous phase obtained from other techniques, thus indication that at high frequency the amorphous phase orientation has a dominant influence on the change of the velocity of the longitudinal acoustic phonons. An anomalous band is present in the spectra of oriented PET films. The angular behavior of this band is provided.

ACKNOWLEDGEMENT

We acknowledge the Office of Naval Research and NSF Polymer Grant No. DMR 79-12457 for providing financial support of this research.

FIGURE CAPTIONS

1. a. Diagram of the goniometer used for angular dependent Brillouin measurements.
- b. Film coordinate axes; stretch direction is along z.
2. Diagram to show the incident, reflected, refracted and scattered rays in the film.
3. Brillouin spectra of the $R_s = 3.41$ film taken at angles: $\alpha = 15^\circ$ and $\alpha = 45^\circ$. R_s is the ratio of the length of the stretched film to that of the unstretched. Note the enhanced contrast of the 5 pass interferometer. The various peaks: (1) quatransverse wave, (2) anomalous Y band (3) quasilongitudinal wave, (4) reflection peak. The spectra were obtained using the VV configuration.
4. Polar plots of sound velocity versus angle in the film. S.D. is the stretch direction.

● - experimental points

— - fit to equation (4)

5. The elastic constants versus draw ratio.

6. Hermann's orientation function parameter f , calculated from the Brillouin results plotted as a function of stretch ratio.

○ our data

◆ data of Ref. 8

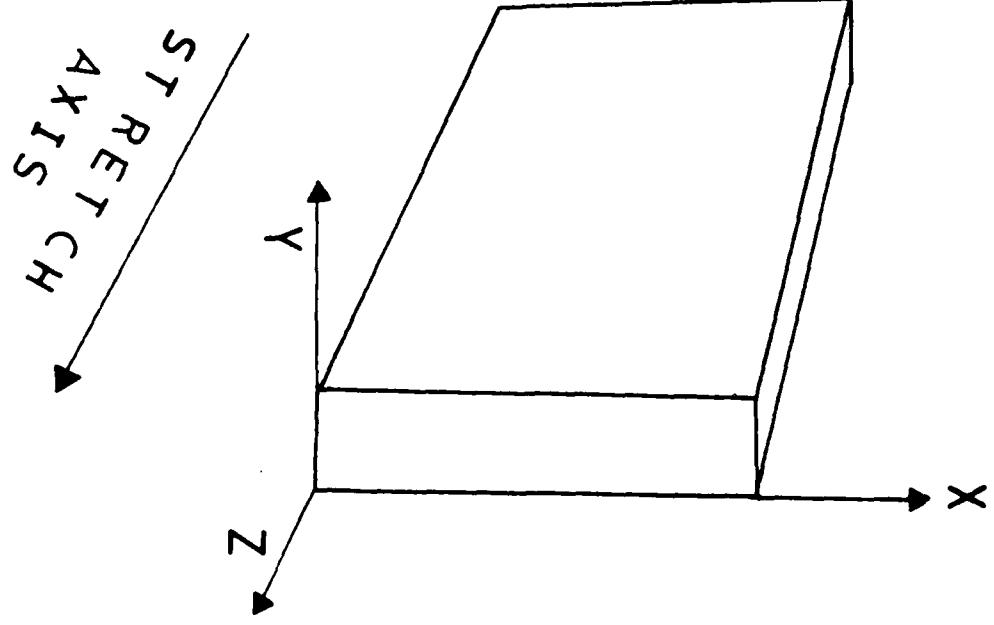
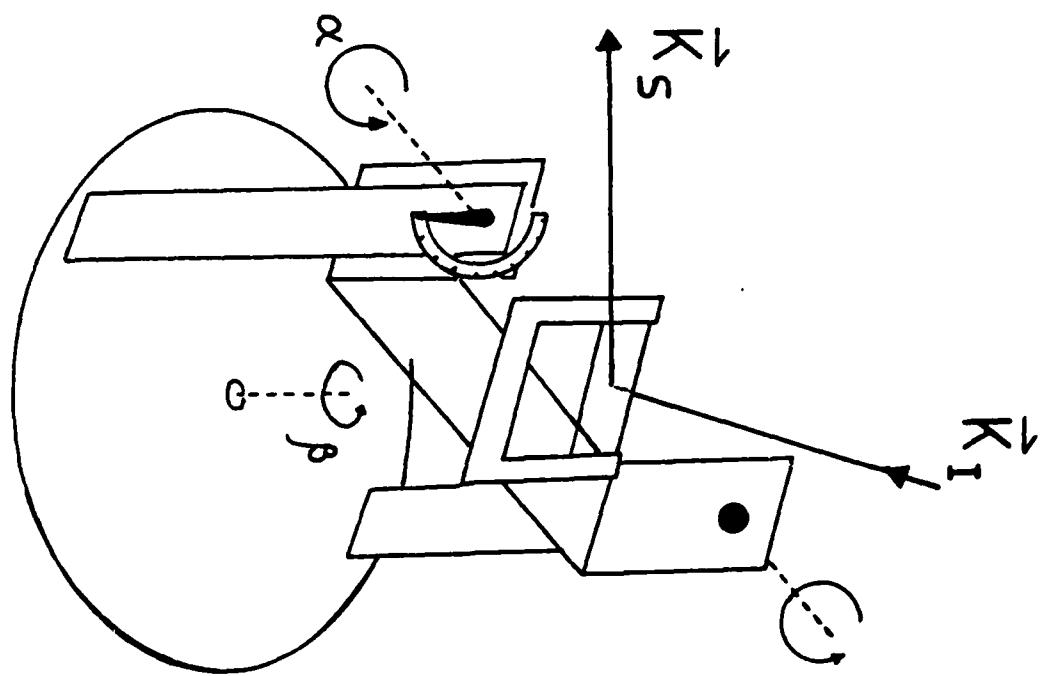
7. Polar plot of the anomalous Y band frequency in the $R_s = 3.57$ film plotted as a function of angle α ; also shown for comparison are the quasilongitudinal frequencies.

● y band

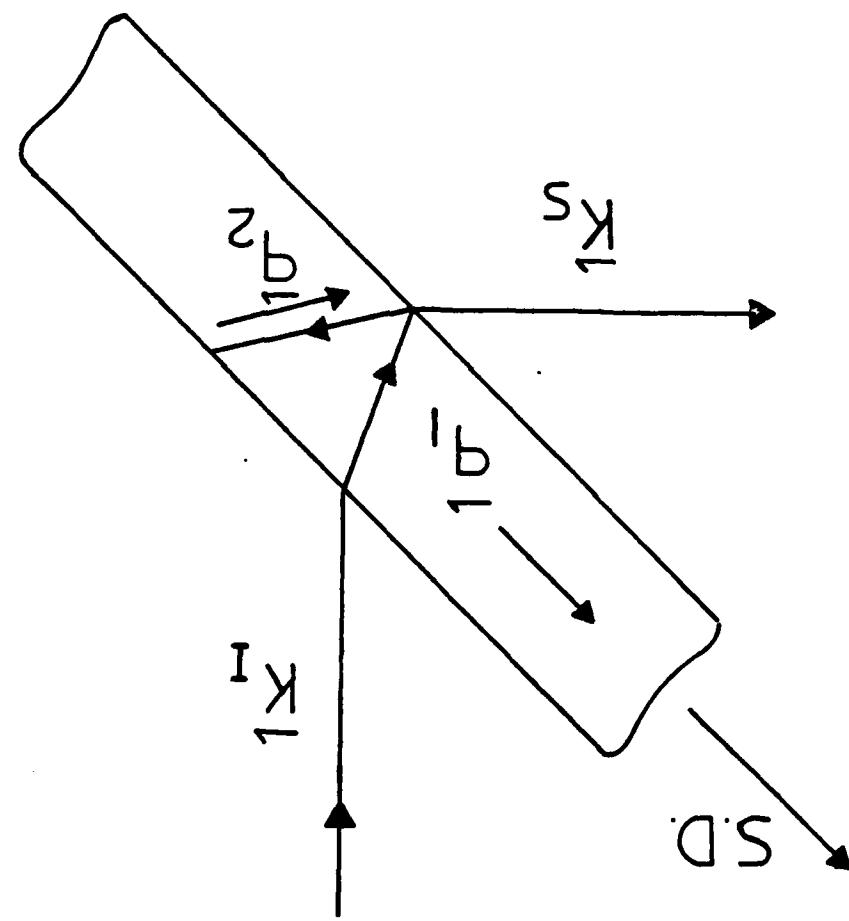
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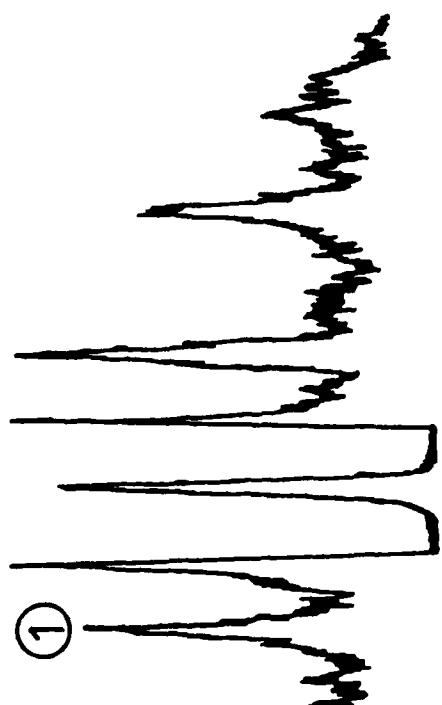
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$$1\sigma = 1s - 1s$$





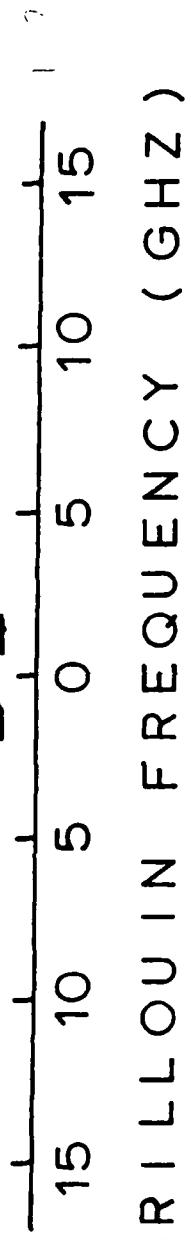
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$\alpha = 45^\circ$



③ ④ ② ①

$\alpha = 15^\circ$

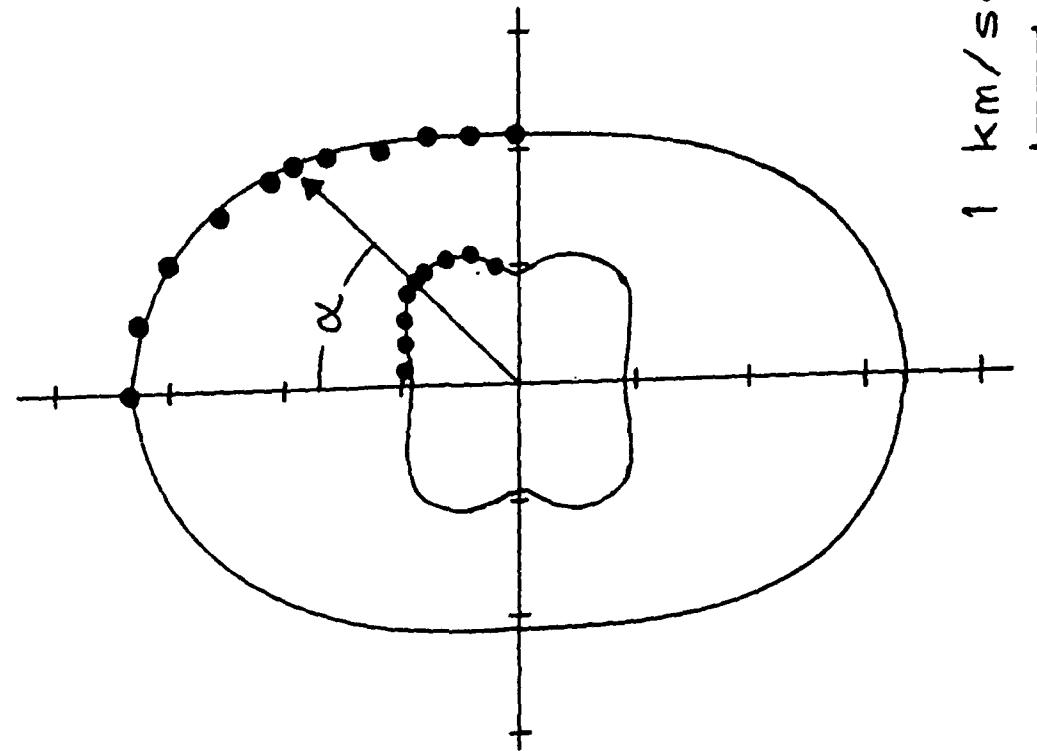


$R_S = 3.57$

$\text{B R I L L O U I N F R E Q U E N C Y (GHz)}$

S.D.

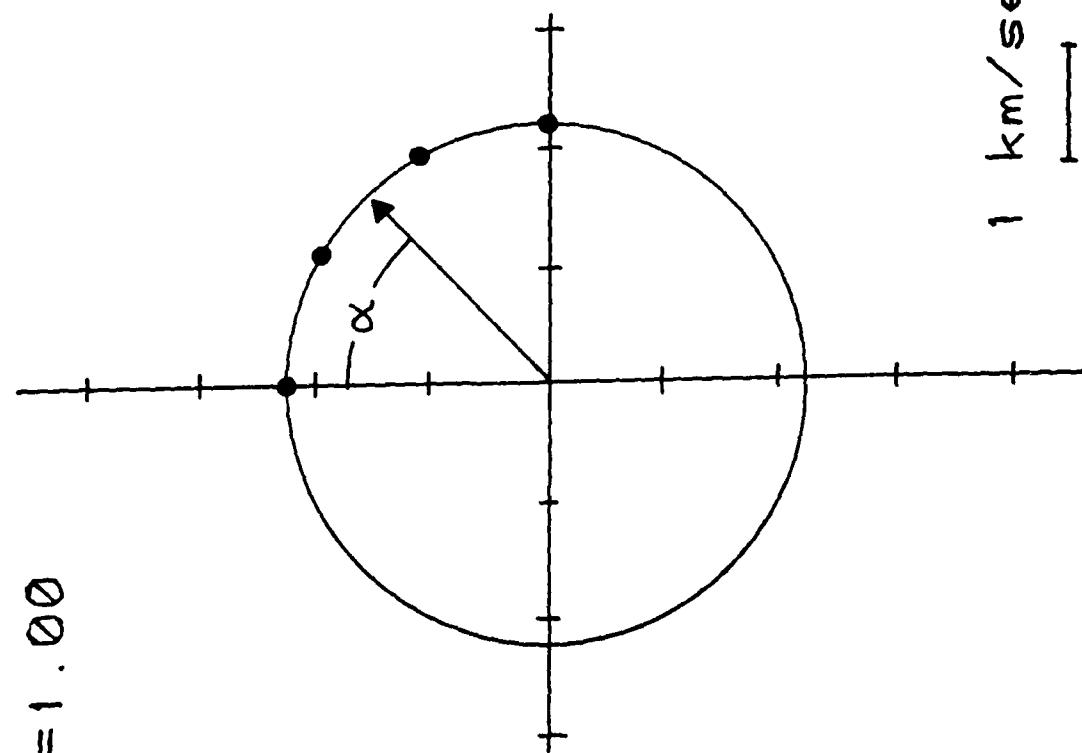
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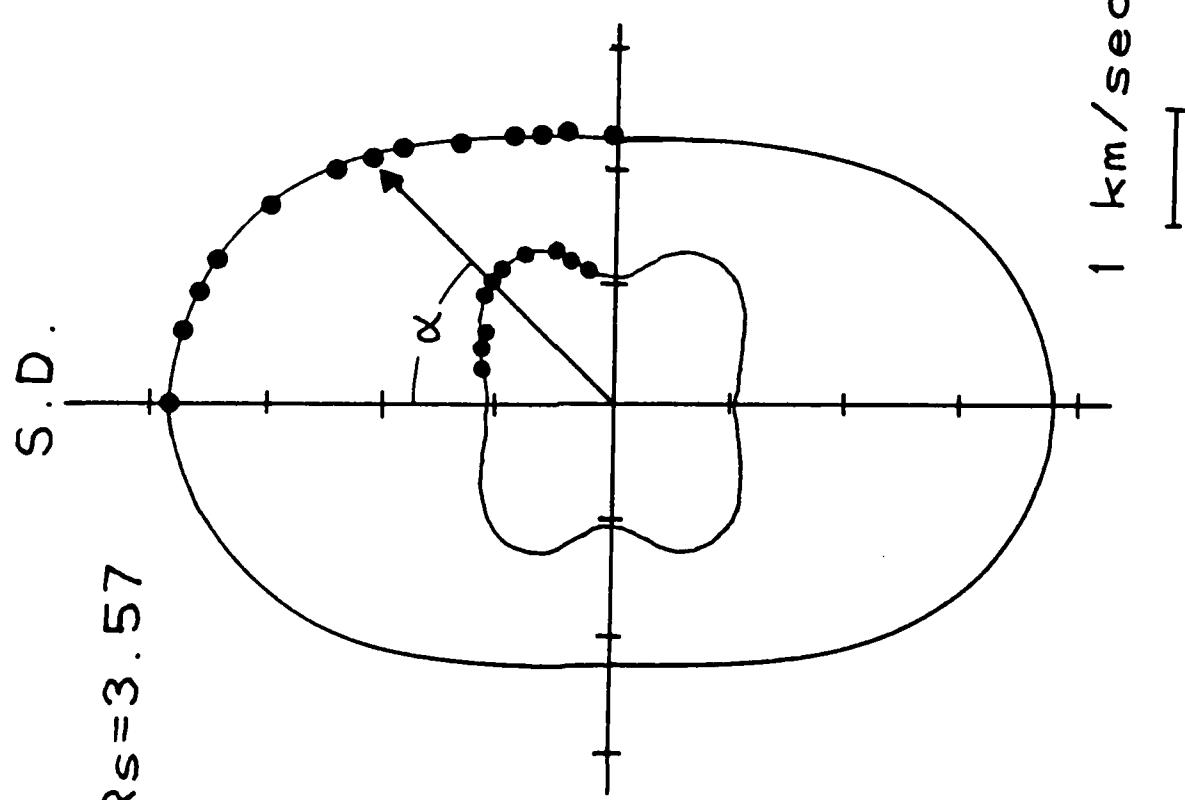
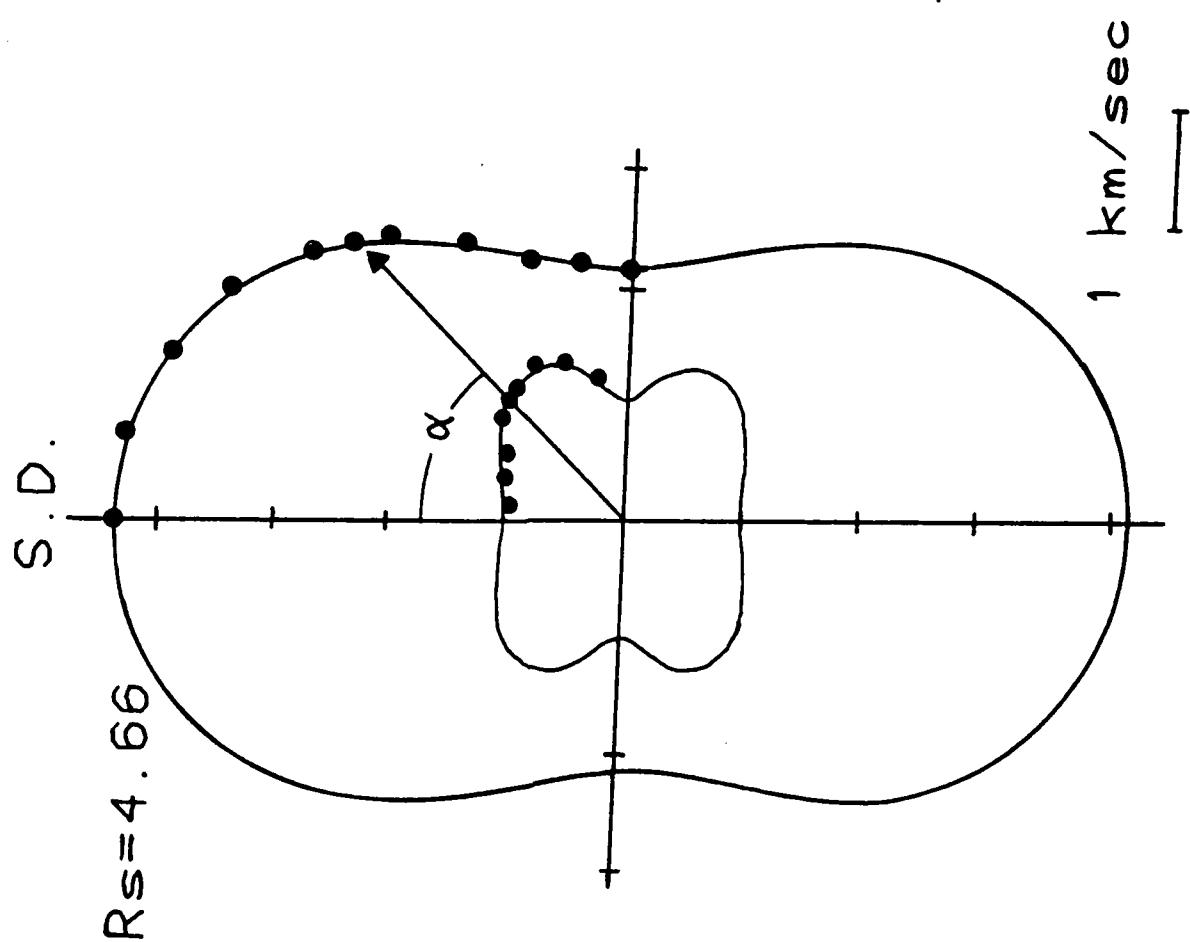


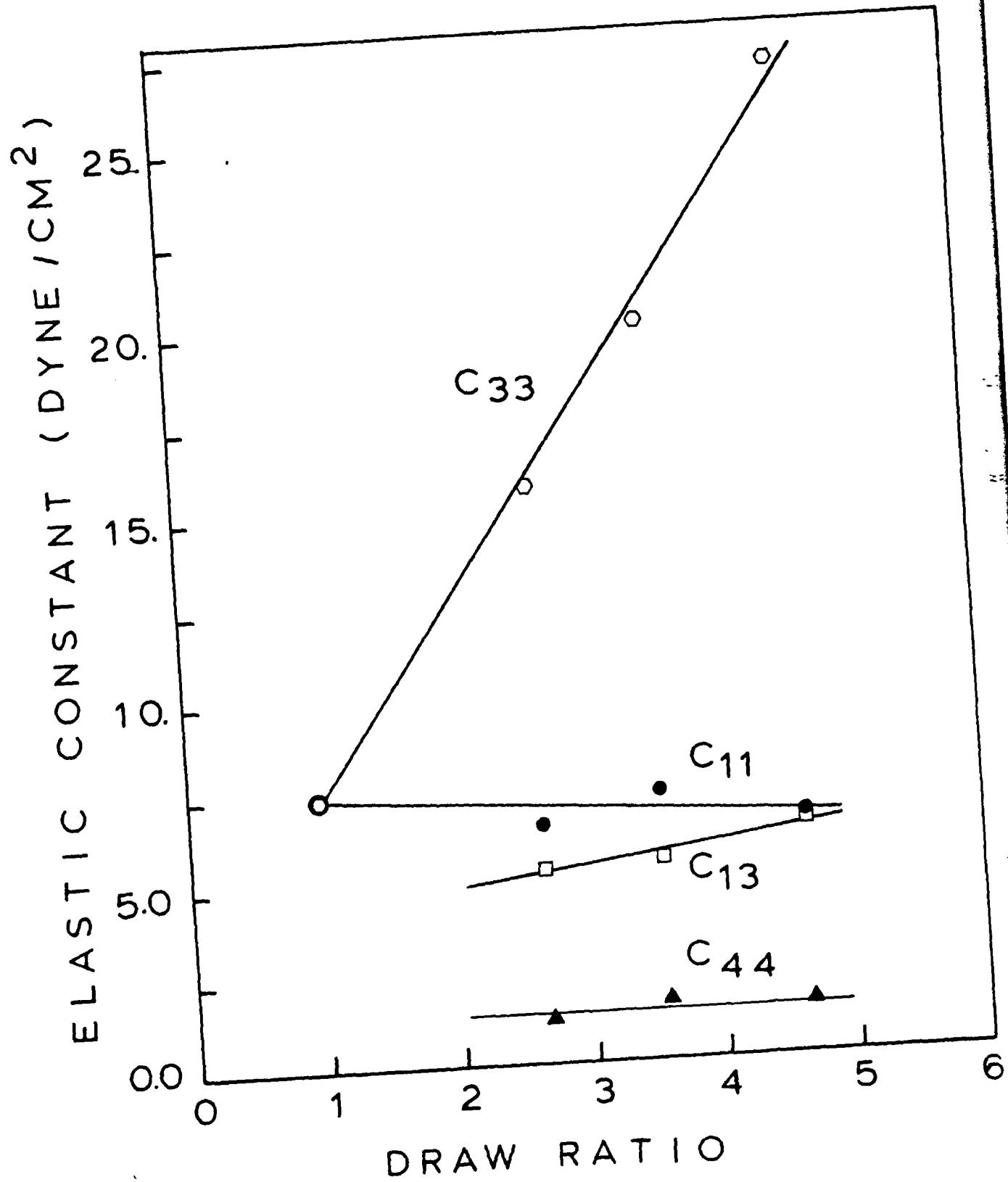
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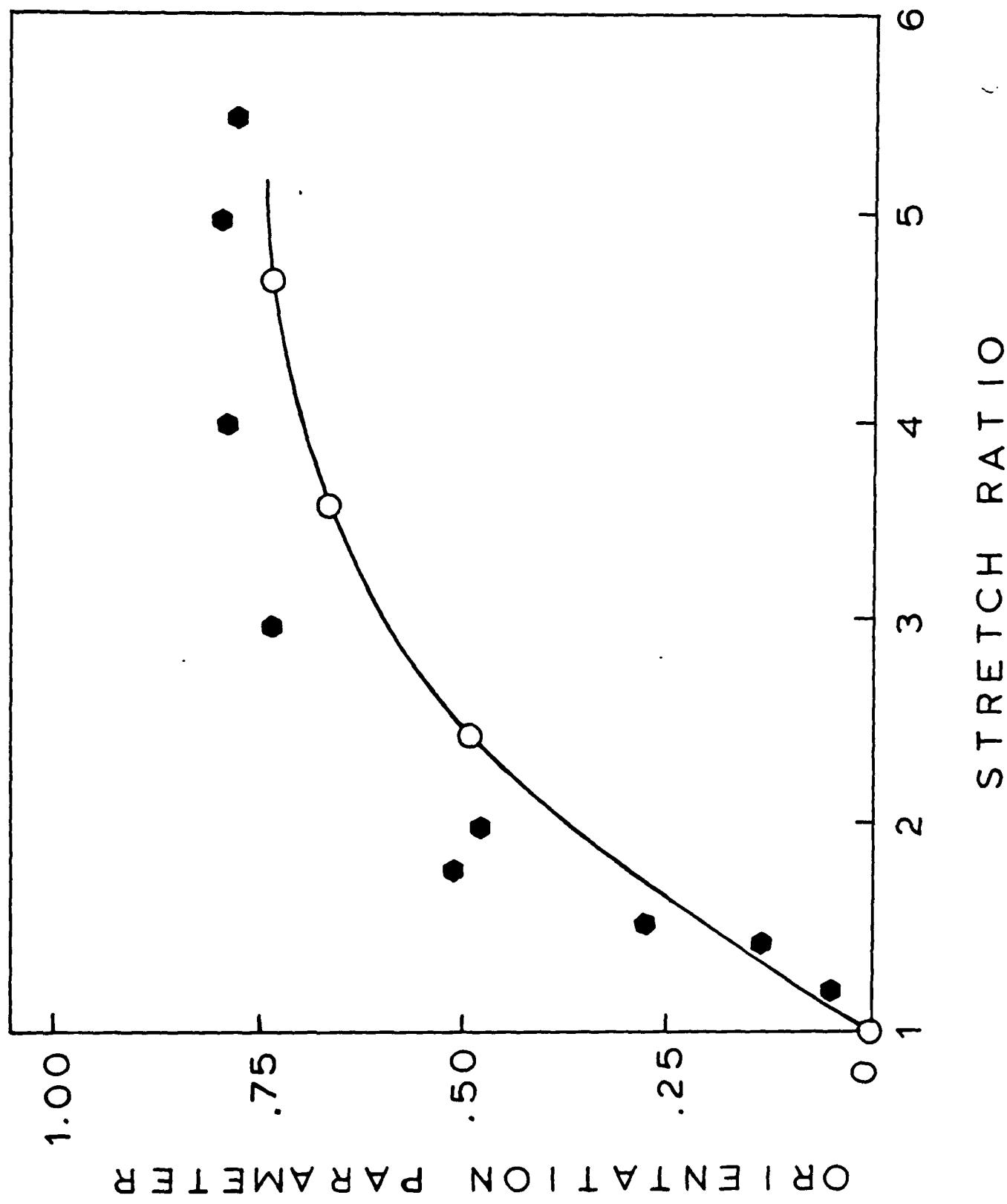
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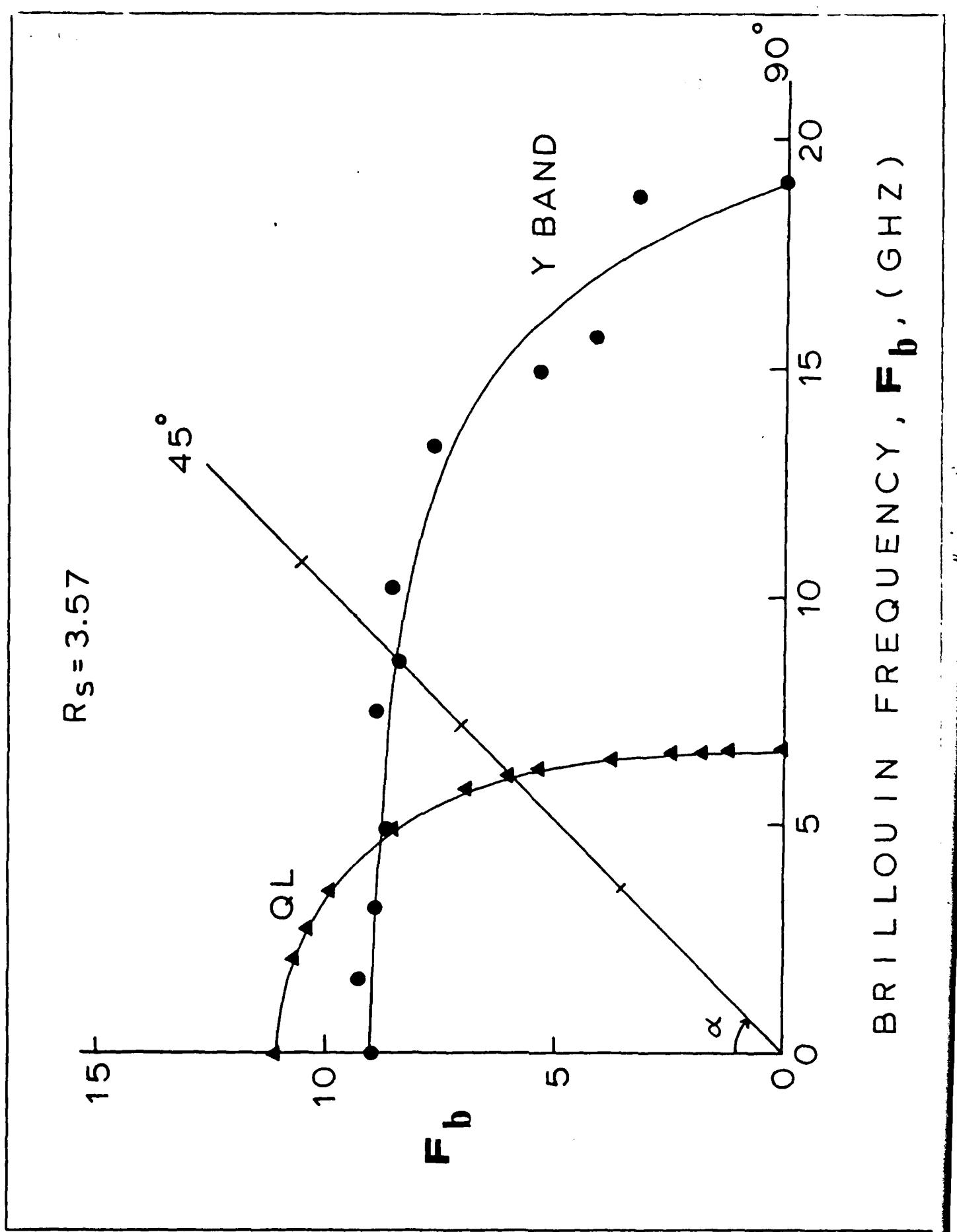
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